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From: Piro, Peter (DPH)
Sent: Tuesday, December 14, 2010 3:07 PM
To: Nassif, Julianne (DPH)
Subject: FW: Refmacal CRM 110 - Holmium Oxide Glass Wavelength Standard

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Hi Julie,
The email below deals with the question of establishing tolerance levels for UV-VIS spectrophotometer. Let me know if you want to pursue the ISO mathematical approach, the less rigorous approach that was discussed below or if you would like to compare both.

From: Stranaska Scientific [mailto:info@stranaska.com]
Sent: Monday, December 13, 2010 2:46 PM
To: 'Piro, Peter (DPH)'
Subject: Refmacal CRM 110 - Holmium Oxide Glass Wavelength Standard

TO: Peter Piro, GC/MS Laboratory Supervisor I, Department of Public Health, Hinton State Laboratory Institute, Forensic Drug Laboratory (Jamaica Plain, MA USA)

Establishing acceptance criteria for the use of a measurement artifact standard such as the CRM 110 in performance evaluation of an analytical instrument is important as you realize. Your work in the forensic lab is very intriguing and something in which I have increasing interest.

The metrological property of measurement uncertainty of the standard must be considered as well as other sources of uncertainty. Unfortunately, I submit that there is no single answer which satisfies every spectrophotometer, every application and every analytical performance requirement (or risk assessment) although there may be a single strategy. The two fundamental questions I would ask myself if I was in your position are the following: how accurate does the measured wavelengths need to be for the given application to facilitate meaningful results, and how defensible are the results?

A more rigorous approach to measurement uncertainty, which helps answer these questions, requires the identification of the difference sources of errors contributing to the overall uncertainty. You have already identified two major sources: the uncertainty in the accuracy of the wavelength scale of the spectrophotometer and the uncertainty in the assigned reference values of the measurement artifact standard (CRM 110) used to establish performance-based metrological traceability and to quantify bias. Temperature as you mentioned in Question 2 is another source. Our expanded uncertainty accounts for only minor temperature variations (22±1°C) so you may want to add some contribution to allow for unknown variation due to your larger temperature swing.

Another important factor is the wavelength uncertainty of your reference spectra used for comparison in the event that your spectra do not overlap or that absorbance maxima for a given absorption band differ. If you do not have an in-house reference standard for comparison, then you need to consider possible inter-instrument bias, that is, difference in scales between your instrument and the instrument used to generate the published data or spectrum.

Once the sources of uncertainty are determined, I recommend the ISO method for combination of their quantified values. Each contribution is mathematically treated as a standard deviation described by an assumed probability distribution function to give an individual standard uncertainty. The individual

standard uncertainties are squared, summed and square rooted to give a combined standard uncertainty. The combined standard uncertainty is then multiplied by a coverage factor (e.g., 2 for a 95% confidence level) to give an expanded uncertainty of the accuracy of the instruments wavelength scale.

I hope this gives you some additional insight on how to proceed. If you feel that it would be beneficial for me to become more involved in providing the necessary metrological perspective, please do not hesitate to contact me for further discussion.

Best regards,

Jerry

Jerry D. Messman, Ph.D.
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-----Original Message-----
From: Piro, Peter (DPH) [mailto:Peter.Piro@state.ma.us]
Sent: Friday, December 10, 2010 9:12 AM
To: 'info@stranaska.com'
Subject: Holmium Oxide Wavelength Calibration Check

Hi Jerry,

Thank you for returning my phone call about our UV. We purchased the Refmacal Holmium Oxide Glass Wavelength Standard for our monthly QC check. While reading the instructional guideline for the use of CRM 110, step 2 talks about establishing the pass/fail acceptance criterion for the wavelength calibration check. I essentially have two questions.

1. Is there a typical way labs (forensic in particular) establish wavelength tolerance when using the holmium oxide standard to check the instrument's wavelength calibration? Our instruments stated wavelength accuracy is +/- 0.5 nm and the holmium oxide uncertainty is roughly +/- 0.3 nm. Our laboratory screens certain drugs using

the UV and relies on published UV data for comparative purposes. Would using the instrument in this manner, without standards, necessitate lower tolerance levels and more frequent calibrations?

2. Are there any other contributing factors laboratories consider when establishing wavelength tolerance? Temperature swings in our lab could easily be +/- 10 degrees Fahrenheit.

Thank you for your assistance

Peter Piro
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